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Fast 5d-4f luminescence in Pr^{3+}-doped $K_3Lu(PO_4)_2$

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ABSTRACT

The double phosphate $K_3Lu(PO_4)_2$ doped with Pr^{3+} ions was prepared by solid-state reaction. The material obtained was characterized by X-ray powder diffraction (XRPD). The luminescence spectroscopy and the excited state dynamics of this material were studied for the first time upon excitation with UV-VUV synchrotron radiation (SR). The material has shown efficient and fast Pr^{3+} 5d-4f emission upon both intra-center and band-gap excitation. A strong dependence of the luminescence spectra as well as the decay kinetics on the temperature was observed due to crystallographic structural phase changes of the $K_3Lu(PO_4)_2$ host.

1. Introduction

The search for new effective scintillator materials capable to convert high-energy radiation (from a few KeV to several MeV) into UV and visible light has been progressed for more than five decades and nowadays constitutes one of the most active research sectors in the field of the radiation detectors. In particular, the rapid evolution in nuclear medical imaging techniques such as computed tomography (CT) and positron emission tomography (PET) has triggered an increased demand for fast radiation detectors [1,2] in order to improve the resolution of tomographic image. Most of the scintillators presently used in CT and PET scanners are based on materials activated with Ce^{3+} ion, which exhibits efficient and relatively fast (~20–70 ns) 5d-4f electric–dipole emission in a broad spectral range (350–600 nm) [3]. Recently much attention has been turned on the study of the 5d-4f emission of Pr^{3+} ions in wide band-gap compounds. The emitting 5d state of Pr^{3+} ions is located at about 12240 cm^{-1} higher energy than that for Ce^{3+} in the same host [4] and its emission lifetime is about 2–3 times shorter [5] allowing faster time responses for scintillators. As a result, the choice of the Pr^{3+} ion as an activator ion for wide band-gap hosts may constitute a promising alternative to the Ce^{3+} one in the development of new scintillator materials for applications requiring time-correlated registration of photons or working at higher count rate.

Our search for novel materials showing fast 5d-4f emission of Pr^{3+} [6,7] has led us to the synthesis and investigation of the time-resolved spectroscopic properties of the double phosphate $K_3Lu(PO_4)_2$ (KLuP) doped with this ion. KLuP is characterized by a high effective atomic number of ~44.5, that is higher than one for $Y_3Al_5O_{12}$ (29.5) or $YAlO_3$ (31.4) widely used as scintillator materials, and potentially advantageous for X- and γ-rays detection. This host crystallizes with a trigonal unit cell, space group *P*-3, density 3.9 g/cm^3 at RT [8]. In this case, the lutetium ion is six-coordinated by the oxygen atoms of the phosphate groups with two non-equivalent sites in the lattice. For this host material two phase transition are known to occur at lower temperature [9]. The first transition occurs at ~250 K and a monoclinic phase ($P2_1/m$ space group) is obtained. The Lu ion retains the six-fold coordination but it occupies only one crystal site. The second phase transition occurring at ~140 K leads to a crystal structure with the same monoclinic space group but coordination of the Lu ion changes to a 7-fold one.

The absorption edge of KLuP is expected to be localized at rather high energy as shown by several double phosphates constituted of lanthanide ions (E_g=7.3–9.5 eV) [6,10] making this compound suitable as efficient host for various optical applications including fast scintillators and vacuum ultraviolet phosphors. To the best of our knowledge, the time-resolved spectroscopy and excited state dynamics of Pr^{3+} ion in KLuP upon VUV excitation with synchrotron radiation has not been reported in literature so far although the Pr^{3+} 5d-4f emission in KLuP:Pr^{3+} powders has been demonstrated in a recent paper [11]. It is also worth to mention that some scintillating and luminescent properties of Ce^{3+} in KLuP single crystals as well as in other double phosphate of $A_3Lu(PO_4)_2$-type (A=Li, Rb, Cs) have been thoroughly studied elsewhere [9,12,13]. Meanwhile some RT VUV spectroscopic properties of Pr^{3+}-doped $ALuP_2O_7$-type alkali lutetium diphosphates (A=Na, K, Rb, and Cs), that are other compounds in the same Lu_2O_3–A_2O–P_2O_5 system, have been investigated in [14] to demonstrate an efficient host-to-Pr^{3+} energy transfer.

In this contribution the synthetic procedure and structural characterization of KLuP doped with Pr^{3+} ion are described in

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detail. Time-resolved emission and excitation spectra as well as luminescence decay curves at different temperatures measured upon selective excitation with synchrotron radiation (SR) in the UV-VUV region are presented and analyzed. The prospective applications of this luminescent material are discussed.

2. Experimental details

Polycrystalline sample of KLuP doped with 1 mol% of Pr^{3+} (substituting for Lu^{3+}) were synthesized using a solid state reaction from K_2CO_3, $(NH_4)_2HPO_4$ (all reagent grade), Lu_2O_3 (Aldrich, 99.99%) and Pr_6O_{11} (Aldrich, 99.999%). Appropriate amounts of the reagents were mixed and heat treated in a horizontal furnace in air for 4 h at 600 °C and 1 h at 950 °C with intermediate regrinding. In between investigations the samples were stored at RT in dark and dry conditions.

X-ray power diffraction patterns (XRPD) were measured with a Thermo ARL X′TRA powder diffractometer, operating in the Bragg–Brentano geometry and equipped with a Cu-anode X-ray source (K_α, $\lambda = 1.5418$ Å) and a Si(Li) Peltier cooled solid state detector. The XRPD patterns were collected with a scan rate of 0.4°/s and an exposure time of 0.72 s in the 5–90° 2θ range. The phase identification was performed with the PDF-4+ 2011 database supplied by the International Centre for Diffraction Data (ICDD). Polycrystalline samples were ground in a mortar and then put in a low background sample holder for the data collection.

Preliminary synchrotron X-ray diffraction powder patterns of an undoped KLuP sample at RT and 220 K were collected in transmission geometry at the MCX beamline located at the multidisciplinary Synchrotron Light Laboratory (ELETTRA) in Trieste (Italy).

Differential scanning calorimetry (DSC) experiments were performed to follow the phase behavior of the sample under investigation using a Q1000, TA Instruments at a heating rate of 10 °C/min. The samples were sealed in aluminum DSC pan for the measurement.

Time-resolved VUV spectroscopic measurements were carried out using the SUPERLUMI facility at HASYLAB of DESY (Hamburg, Germany) using synchrotron radiation (SR) from the DORIS III storage ring as an excitation source. For the selective excitation and measurements of excitation spectra in the range of 3.7–20.4 eV a 2 m monochromator in McPherson mounting with a resolution of 3.2 Å was used. The detection of the luminescence was performed with a 0.3 m ARC SpectraPro-308i monochromator equipped with a high-speed R3809U-50S (Hamamatsu) microchannel plate (MCP) detector. The time-resolved spectra were recorded within two time gates (TGs): 2–11 ns (fast time gate) and 50–61 ns (slow time gate) relative to the beginning of the SR pulse. The time-integrated spectra were recorded counting the emission signal within the whole time period of 96 ns available between SR pulses at a 10 bunch mode (BM) of the storage ring. The measurements were performed in the ultra-high-vacuum chamber ($\sim 10^{-9}$ mbar) in the temperature range of 8–300 K. The excitation spectra were corrected for the wavelength-dependent variation of the SR intensity using the sodium salicylate signal. A background signal corresponding to the dark count of the MCP detector was subtracted from the original spectra and decay curves.

3. Results and discussion

3.1. Structural characterization

The structural characterization at RT of the synthesized Pr^{3+} doped sample was performed by means of X-ray powder diffraction (XRPD). All the diffraction peaks in the XRPD pattern of Pr^{3+}-doped KLuP sample are compatible with ICDD data of the trigonal (*P*-3 space group) KLuP (PDF Card no. 01-085-1586) (Fig. 1). No other phases are detected indicating that the sample synthesized is a single phase. In addition, we observed a small expansion of the cell volume as a consequence of substitution of Lu^{3+} ion (ionic radius 0.861 Å, in six-fold coordination [15]) with larger Pr^{3+} one (0.99 Å, in six-fold coordination [15]). The cell volume of the Pr^{3+} doped sample and the undoped one are 617.07(1) $Å^3$ and 616.66(1) $Å^3$, respectively.

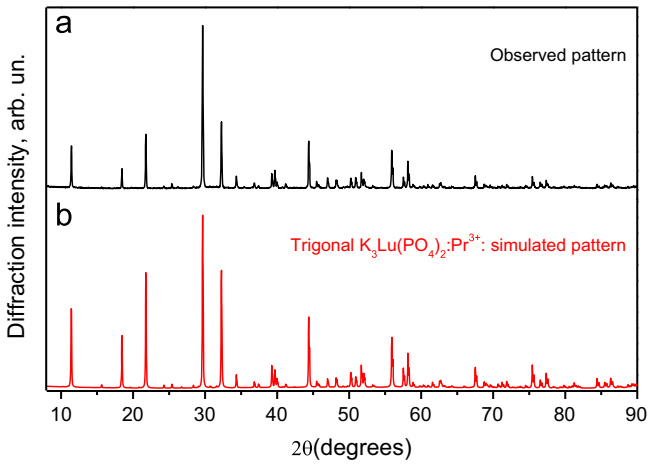

Fig. 1. Experimental XRPD pattern of the KLuP:1% Pr^{3+} powder at 300 K (a) and simulated XRPD pattern of the trigonal KLuP (PDF card number 01–085-1586) (b).

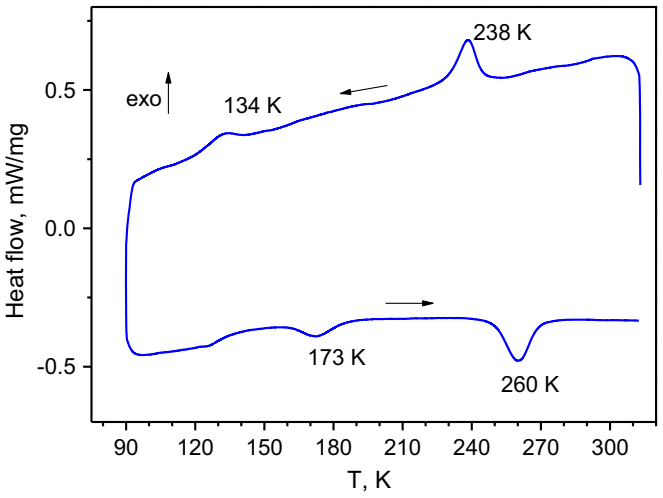

Fig. 2. DSC scan profile of KLuP:Pr^{3+} powders. Both scan rates (up and down) are 2 K/min.

A supplementary structural characterization has been performed by means of the Rietveld refinement on the synchrotron X-ray powder diffraction pattern. The corresponding results will lie the groundwork of another paper. For the present paper an important issue worthy to be mentioned is that, on average, the Lu–O bond lengths in the case of trigonal phase are Lu(1)–O = 2.16(1) Å and Lu(2)–O = 2.14(1) Å, whereas in the case of the monoclinic phase, stable in the temperature range 140–250 K, the average Lu–O bond length for the only available Lu^{3+} crystal site seems to be longer than in the case of the trigonal one. The Rietveld refinement on the powder pattern of the low-temperature monoclinic phase is only preliminary.

The differential scanning calorimetry (DSC) of KLuP:Pr^{3+} sample is shown in Fig. 2. The DSC scan decreasing the temperature shows distinct exothermic peaks at approximately $T_1 = 238$ K and $T_2 = 134$ K associated with the two phase transitions documented

in [9]. These crystal structural transformations appears to be fully reversible even though the scan increasing the temperature evidences a slight hysteresis of a few degrees for both transitions (T_1=260 K and T_2=173 K).

3.2. Luminescence spectroscopy and dynamics

Fig. 3 gathers the RT time-resolved and time-integrated emission spectra of KLuP:Pr^{3+} recorded upon direct intra-center and host excitation respectively. The RT spectra of KLuP:Pr^{3+} obtained upon direct intra-center excitation at 190 nm (Fig. 3a) are dominated by a broad band extending from 225 and 375 nm and centered at about 275 nm. This band is well pronounced in the fast TG spectrum and it is assigned to the parity-allowed interconfigurational transitions from the lowest excited 5d state to the states of the ground $4f^2$ electronic configuration of Pr^{3+} ion ($4f^15d^1 \rightarrow 4f^2$). The tail observed on the long wavelength side of the emission indicates the presence of another emission overlapping with the dominant Pr^{3+} emission, consistent with the availability of two distinct crystallographic sites for Pr^{3+} in the RT trigonal KLuP phase [9].

Fig. 3b presents the RT time-integrated and time-resolved spectra recorded upon excitation at 90 nm; this corresponds to transitions far above the band-gap (E_g). A typical bandgap energy for many double phosphates is about 7.3–9.5 eV (170–130 nm) as suggested in [10] by the analysis of VUV reflection spectra. The time-integrated spectrum is similar to that observed upon direct intra-center excitation showing a broad band extending from 225 to 380 nm, most relevant in the fast TG spectrum. A shoulder pronounced at about 325 nm reflects the presence of the two different Pr^{3+} emitting sites which are integrally excited under bandgap excitation. At the same time, the nearly twofold increase of the signal intensity detected within slow TG around the features related to the Pr^{3+} 5d-4f transitions indicates the existence of some delayed host-to-impurity energy transfer processes. Furthermore, it is worth noting that for both direct and host excitations no emission features associated with the 4f-4f transitions from the $^3P_0$ and $^1D_2$ levels are observed. This can be explained on the basis of the Stokes shift (see below).

Fig. 4 collects the time-integrated emission spectra of KLuP:Pr^{3+} recorded at 8, 190 and 300 K upon intra-center excitation at 190 nm. Interestingly, the position and the profile of the Pr^{3+} 5d-4f emission band become significantly different with increasing

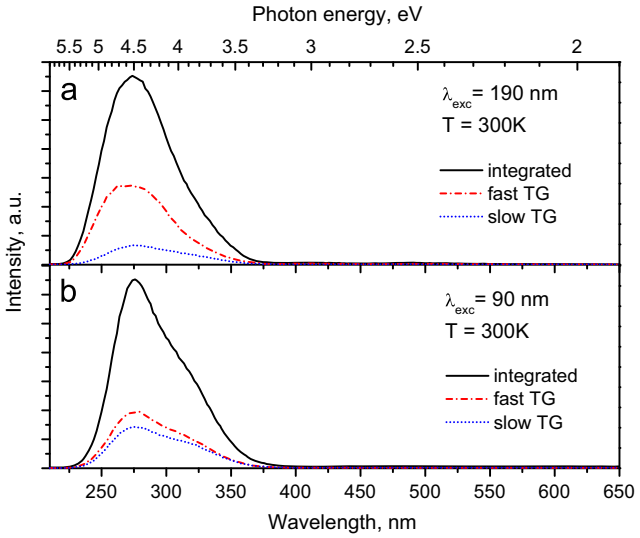

Fig. 3. Time-integrated and time-resolved emission spectra of KLuP:Pr^{3+} powders recorded upon selective excitation at 190 nm (a) and 90 nm (b) at 300 K.

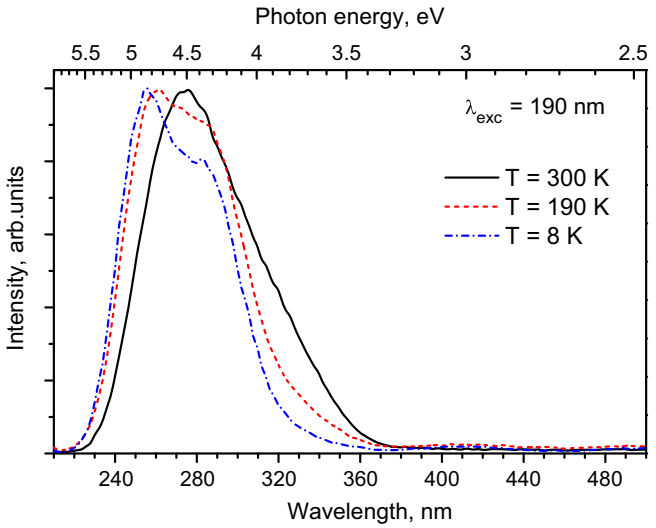

Fig. 4. Time-integrated emission spectra of KLuP:Pr^{3+} powders recorded upon intra-center excitation at 190 nm at 8, 190 and 300 K.

temperature from 8 to 300 K, in agreement with what was observed for KLuP:Ce^{3+} single crystals in [9]. Since in KLuP the dopant ions substitute only for the Lu^{3+} ions, the above-mentioned changes have to be linked with the crystal structure transformations of KLuP and consequently with the local lutetium coordination and symmetry changes. As matter of the fact, the energy of the $4f^15d^1 \rightarrow 4f^2$ emission band strongly depends on both the covalence effect and the crystal field strength induced by the ligands. As a result of covalent bonding between the 5d and the ligand orbitals, the 5d orbitals expand by partial delocalization over the ligands and consequently the energy is lowered (nephelauxetic effect). The red shift of the Pr^{3+} 5d-4f emission band observed with increasing temperature from 8 to 300 K is linked to a possible increase of the covalency of the Lu–O bonds moving from the monoclinic low temperature phase to the trigonal RT one. This proposition correlates with preliminary results of our structural study of synchrotron X-ray powder diffraction suggesting a decrease of the Lu–O bond length when crystal structure changes from the monoclinic to trigonal (see above).

Fig. 5 shows time-integrated and time-resolved excitation spectra of KLuP:Pr^{3+} recorded monitoring emission at 320 nm for RT and at 280 nm for T=8 K. The UV part of the time-integrated excitation spectrum recorded at RT (Fig. 5a) is characterized by a broad structured band consisting of maxima at about 231, 222 nm and low intensity features spread from about 170–210 nm. As can be seen from the time-resolved spectra, the UV band is dominated by a fast decaying emission signal. We assign this structured band to transitions from the ground $4f^2$ state to the various 5d crystal field levels of Pr^{3+} ion (in the two possible Lu sites of the RT trigonal KLuP phase). On the base of data on 4f-5d transition energy for the Ce^{3+} ion in KLuP at RT [9] and known energy shift of $\sim$12240 cm^{-1} between 5d states of Ce^{3+} and Pr^{3+} [4] the first Pr^{3+} 4f-5d transition can be expected at 235 nm, which is in good agreement with the data presented in Fig. 4. The Stokes shift for the RT Pr^{3+} 5d-4f transitions was estimated after suitable deconvolution of the band profiles and resulted to be about 2800 cm^{-1}. This is in agreement with the model for Pr^{3+} emission proposed by Srivastava et al. [11] which suggests that 5d-4f transitions dominate over 4f-4f ones when the Stokes shift is lower than $\sim$3200 cm^{-1}.

At near liquid He temperature the excitation spectra (Fig. 5-b) demonstrate significant change in the spectral range above 160 nm compared to the RT ones reflecting the above-mentioned crystallographic structural phase transformation of KLuP and nephelauxetic

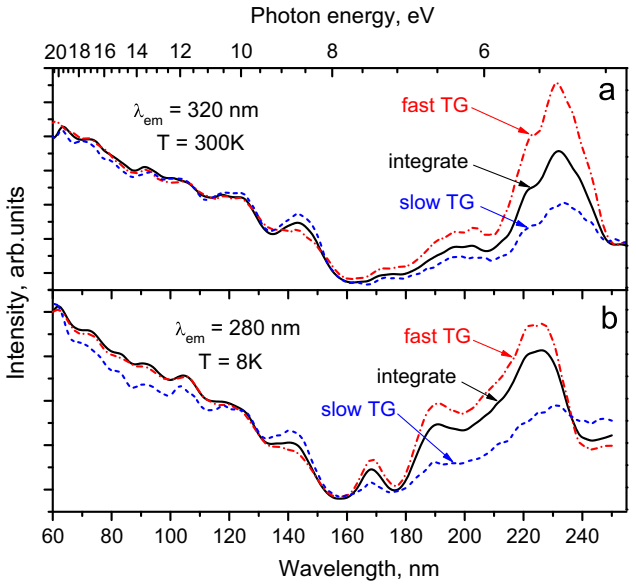

Fig. 5. Time-integrated and time-resolved excitation spectra monitoring 5d-4f emission of Pr^{3+} in KLuP:Pr^{3+} powders at $T=300$ K (a) and $T=8$ K (b).

effect. The lowest energy 4f-5d excitation maximum is observed at about 225 nm being blue-shifted relative to that at RT by about 1100 cm^{-1}. Well pronounced maxima related to higher-energy 4f-5d transitions are observed at about 191 and 168 nm.

Excitation processes related to energy transfer from the host electronic excitations are observed in the excitation spectra below ~160 nm and ~157 nm for RT and $T=8$ K, respectively. This correlates with the bandgap energies reported for similar double phosphates in Ref. [10]. We note that the excitation spectra do not demonstrate any sharp rising excitation feature at beginning of fundamental observation that indicate lack of the excitonic energy transfer mechanism. At the same time, a gradual rise of excitation spectra towards shorter wavelength suggests that host-to-Pr^{3+} energy transfer is dominated by the recombination mechanism. One of the reasons for domination of recombination mechanism may be connected with low energy of relaxed excitons that is insufficient for reabsorption by Pr^{3+} 5d states. We did not observe an exciton emission from our Pr^{3+} doped samples that is probably due to the fact that intrinsic electronic excitations are mostly captured by Pr^{3+} ions. At the same time, we were unable to find any published reports on exciton emission in KLuP. Nevertheless, by analogy with energy of self-trapped exciton emission in other phosphates (~23800 cm^{-1} in YPO_4 [16], ~22600 cm^{-1} in $LiY(PO_4)_3$ [17]) we suppose that in case of KLuP it is very probably located at energy much smaller than that required for 4f-5d absorption of Pr^{3+} in this material (> 43000 cm^{-1}).

The temperature dependent decay curves of the Pr^{3+}5d-4f emission upon both intra-center and bandgap excitations are presented in Fig. 6-a and -b, respectively. Based on the emission spectra recorded at different temperatures, we performed the decay measurements at spectral points best corresponding to Pr^{3+}5d-4f emission in the particular phases: at $\lambda_{em}=320$ nm for Pr^{3+} emission in the trigonal RT phase for $T=300$ and 240 K, and at $\lambda_{em}=280$ nm for Pr^{3+} emission in the monoclinic low temperature phase for $T \leq 190$ K. At $T=8$ K the intra-center excited Pr^{3+}5d-4f emission demonstrates a nearly single exponential decay curve which can be satisfactory fitted with a lifetime $\tau=14.8$ ns. Interestingly, at higher temperatures the decay curves demonstrate a slight contribution from a shorter decay component that is likely due to a partial quenching of the emission by some quenching centers. We note that somewhat similar effect was observed in the decay curves recorded

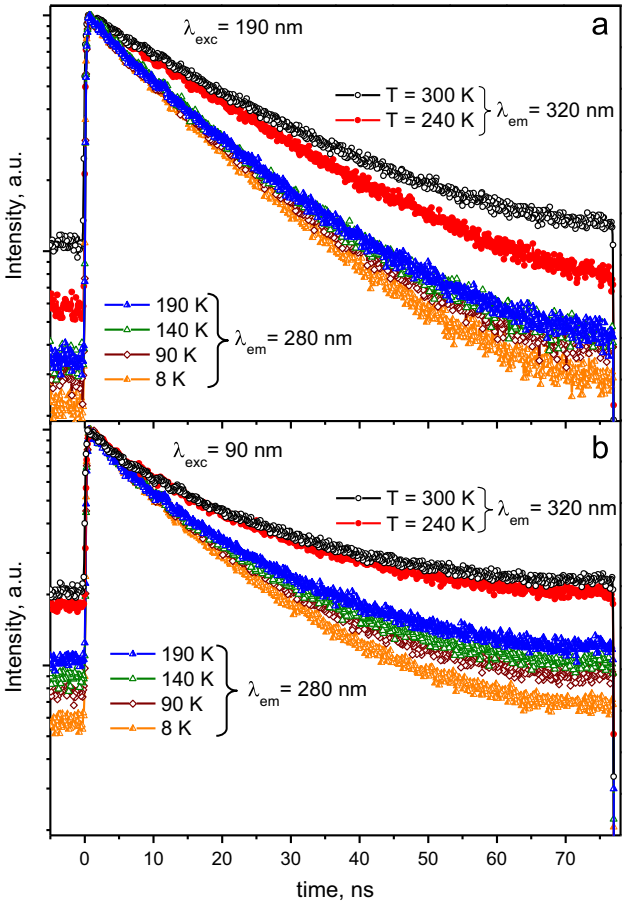

Fig. 6. Decay curves recorded monitoring 5d-4f emission of Pr^{3+} upon intra-center excitation at 190 nm, (a) and host excitation at 90 nm (b) at various temperatures.

for KLuP single crystals and for other double phosphate of $A_3Lu(PO_4)_2$-type (A=Li, Rb, Cs) doped with Ce^{3+} ions [9,12,13]. In addition, the emission decay profiles become progressively longer with temperature increasing, that correlates with the previously observed shift of the Pr^{3+}5d-4f emission band toward longer wavelengths (Fig. 4). To qualitatively analyze the effect of the temperature on the excited state dynamics of Pr^{3+} in KLuP we approximated the non-exponential decay profiles as a sum of two exponential functions and quantified the decay processes by using an average decay time τ_{avg} as suggested in [18]:

$$\tau_{avg} = (A_1\tau_1^2 + A_2\tau_2^2)/(A_1\tau_1 + A_2\tau_2) \quad (1)$$

where τ_1 and τ_2 represent short- and long-lifetimes, respectively, and A_1 and A_2 the corresponding intensity coefficients. The calculated average decay time for the decay curves recorded at 90, 140, 190, 240 and 300 K are summarized in Table 1. The lifetime for the 320 nm emission at RT is a factor 1.5 faster than that observed for 280 nm emission at 8 K. On the basis of a recent analysis carried out by Zych et al. [19], the best agreement between experimental and theoretical Pr^{3+} 5d-4f radiative decay rate is obtained for a λ^3 emission wavelength dependence. Considering that the maximum emission intensity in the RT spectrum is located at 275 nm and the one in the 8 K spectrum is located at 256 nm, a factor of 1.2 is to be expected in our case.

The decay curves recorded upon host excitation at various temperatures (Fig. 6-b) show a significant contribution of a slow decay component (probably with a micro- or millisecond lifetime) presented as a piling-up, that could be related to the presence of defects (afterglow). Nevertheless, the main decay component is found to be characterized by an average decay time very similar to

Table 1
Average decay times for Pr^{3+} 5d-4f luminescence in KLuP at various temperatures upon both intra-center (λ_{exc}=190 nm) and band-gap (λ_{exc}=90 nm) excitation.

T (K)	λ_{em} (nm)	τ_{avg} (ns)	
		λ_{exc}=190 nm	λ_{exc} =90 nm
90	280	15.2	15.1
140	280	15.7	15.6
190	280	16	16.5
240	320	19.4	19.5
300	320	20.3	19.9

that calculated for the curves recorded upon direct excitation (see Table 1). Furthermore, it is important to note that the decay curves do not display any significant rise of the emission intensity just after the excitation pulse indicating that the excitation energy is very rapidly transferred to the emitting centers; this latter is a crucial requirement to produce a good scintillator material for applications such as those based on time-correlated detection of photons.

4. Conclusions

In summary, KLuP doped with Pr^{3+} ions have been synthesized by a solid state reaction. The time-resolved VUV luminescence spectroscopic properties of the material have been studied using synchrotron radiation. Upon both direct intra-center and band-gap excitation the emission of KLuP:Pr^{3+} is dominated by Pr^{3+} 5d-4f interconfigurational radiative transitions. The VUV excitation spectra clearly demonstrated a high efficiency of the host-to-Pr^{3+} energy transfer in KLuP. Luminescence spectra as well as decay curves displayed significant changes with the temperature within the range 8–300 K reflecting the crystal phase transitions of this compound documented in [9]. The decay curves of the 5d-4f emission of Pr^{3+} recorded upon both direct and band-gap excitation at 8 K are characterized by decay times of about 15 ns. The increase of the lifetimes up to about 20 ns evidenced at RT correlates with shift of the Pr^{3+} 5d-4f emission towards longer wavelength and is determined by the phase changes followed by increase of the covalency in the Pr-O bonds.

Finally, the VUV spectroscopic properties of KLuP:Pr^{3+} along with its high effective atomic number revealed good potentialities for application of this material as a fast scintillator. To support the latter, additional experimental studies of scintillating properties such as energy resolution, light yield and decay time upon excitation with pulsed X- or gamma-ray sources are required.

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